

(19)



Europäisches Patentamt  
European Patent Office  
Office européen des brevets



(11)

**EP 1 001 133 A1**

(12)

**EUROPEAN PATENT APPLICATION**

(43) Date of publication:  
17.05.2000 Bulletin 2000/20

(51) Int Cl.7: **E21B 43/26, E21B 43/267,  
E21B 43/04**

(21) Application number: **99307988.8**

(22) Date of filing: **11.10.1999**

(84) Designated Contracting States:  
**AT BE CH CY DE DK ES FI FR GB GR IE IT LI LU  
MC NL PT SE**  
Designated Extension States:  
**AL LT LV MK RO SI**

(30) Priority: **09.11.1998 US 188880**

(71) Applicant: **Halliburton Energy Services, Inc.  
Duncan, Oklahoma 73536 (US)**

(72) Inventors:  
• **Weaver, Jim D.  
Duncan, Oklahoma 73533 (US)**

• **Stanford, James R.  
Duncan, Oklahoma 73533 (US)**  
• **Nguyen, Phillip D.  
Duncan, Oklahoma 73533 (US)**  
• **Dewprashad, Brahmadeo T.  
Norman, Oklahoma 73069 (US)**

(74) Representative: **Waln, Christopher Paul et al  
A.A. Thornton & Co.  
235 High Holborn  
London WC1V 7LE (GB)**

(54) **Treating subterranean formation**

(57) A subterranean formation is treated with a particulate laden fluid whereby particulate flowback is reduced or prevented. The fluid comprises a mixture of a particulate, a tackifying compound and a multifunctional material, and the tackifying compound retards move-

ment of at least a portion of the particulate within the formation upon flow of fluids from the subterranean formation. The multifunctional material subsequently reacts with said tackifying compound to consolidate at least a portion of said particulate within said formation.

**EP 1 001 133 A1**

## Description

[0001] This invention relates to a method of treating a subterranean formation and, more particularly, to a method and means for controlling particulate solids transport during the production of hydrocarbons from a subterranean formation.

[0002] Transport of particulate solids during the production of hydrocarbons from a subterranean formation is a continuing problem. The transported solids can erode or cause significant wear in the hydrocarbon production equipment used in the recovery process. The solids also can clog or plug the wellbore thereby limiting or completely stopping fluid production. Further, the transported particulates must be separated from the recovered hydrocarbons adding further expense to the processing.

[0003] The particulates which are available for transport may be present due to an unconsolidated nature of a subterranean formation and/or as a result of well treatments placing particulates in a wellbore or formation, such as, by gravel packing or propped fracturing.

[0004] In the treatment of subterranean formations, it is common to place particulate materials as a filter medium and/or a proppant in the near wellbore area and in fractures extending outwardly from the wellbore. In fracturing operations, proppant is carried into fractures created when hydraulic pressure is applied to these subterranean rock formations to a point where fractures are developed. Proppant suspended in a viscosified fracturing fluid is carried outwardly away from the wellbore within the fractures as they are created and extended with continued pumping. Upon release of pumping pressure, the proppant materials remain in the fractures holding the separated rock faces in an open position forming a channel for flow of formation fluids back to the wellbore.

[0005] Proppant flowback is the transport of proppants back into the wellbore with the production of formation fluids following fracturing. This undesirable result causes undue wear on production equipment, the need for separation of solids from the produced hydrocarbons and occasionally also decreases the efficiency of the fracturing operation since the proppant does not remain within the fracture and may limit the width or conductivity of the created flow channel. Proppant flowback often may be aggravated by what is described as "aggressive" flowback of the well after a stimulation treatment. Aggressive flowback generally entails flowback of the treatment fluid at a rate of from about 0.001 to about 0.1 barrels per minute (BPM) per perforation of the treatment fluids which were introduced into the subterranean formation. Such flowback rates accelerate or force closure of the formation upon the proppant introduced into the formation. The rapid flowrate can result in large quantities of the proppant flowing back into the wellbore before closure occurs or where inadequate bridging within the formation occurs. The rapid flowback is highly desirable for the operator as it returns a wellbore to production of hydrocarbons significantly sooner than would result from other techniques.

[0006] Currently, the primary means for addressing the proppant flowback problem is to employ resin-coated proppants or resin consolidation of the proppant which are not capable of use in aggressive flowback situations. Further, the cost of resin-coated proppant is high, and is therefore used only as a tail-in in the last five to twenty five percent of the proppant placement. Resin-coated proppant is not always effective since there is some difficulty in placing it uniformly within the fractures. Another means showing reasonable effectiveness has been to gradually release fracturing pressure once the fracturing operation has been completed so that fracture closure pressure acting against the proppant builds slowly allowing the proppant particles to stabilize before flowback of the fracturing fluid and the beginning of hydrocarbon production. Such slow return is undesirable, however, since it reduces the production from the wellbore until the treatment fluid is removed.

[0007] In unconsolidated formations, it is common to place a filtration bed of gravel in the near-wellbore area in order to present a physical barrier to the transport of unconsolidated formation fines with the production of hydrocarbons. Typically, such so-called "gravel packing operations" involve the pumping and placement of a quantity of gravel and/or sand having a mesh size between about 10 and 60 mesh on the U.S. Standard Sieve Series into the unconsolidated formation adjacent to the wellbore. It is sometimes also desirable to bind the gravel particles together in order to form a porous matrix through which formation fluids can pass while straining out and retaining the bulk of the unconsolidated sand and/or fines transported to the near wellbore area by the formation fluids. The gravel particles may constitute a resin-coated gravel which is either partially cured and subsequently completes curing or can be cured by an overflush of a chemical binding agent once the gravel is in place. It has also been known to add various hardenable binding agents or hardenable adhesives directly to an overflush of unconsolidated gravel in order to bind the particles together.

[0008] U. S. Patents 5,330,005, 5,439,055 and 5,501,275 disclose a method for overcoming the difficulties of resin coating proppants or gravel packs by the incorporation of a fibrous material in the fluid with which the particulates are introduced into the subterranean formation. The fibers generally have a length ranging upwardly from about 2 millimeters and a diameter of from about 6 to about 200 microns. Fibrillated fibers of smaller diameter also may be used. The fibers are believed to act to bridge across constrictions and orifices in the proppant pack and form a mat or framework which holds the particulates in place thereby limiting particulate flowback. The fibers typically result in a 25 percent or greater loss in permeability of the proppant pack that is created in comparison to a pack without the fibers.

[0009] While this technique may function to limit some flowback, it fails to secure the particulates to one another in

the manner achieved by use of resin coated particulates.

[0010] U.S. Patent 5,501,274 discloses a method for reducing proppant flowback by the incorporation of thermoplastic material in particulate, ribbon or flake form with the proppant. Upon deposition of the proppant and thermoplastic material in the formation, the thermoplastic material softens and causes particulates adjacent the material to adhere to the thermoplastic creating agglomerates. The agglomerates then bridge with the other agglomerates and other particulates to prevent flowback from the formation.

[0011] It would be desirable to provide a more permanent method which will bind greater numbers of particles of the particulate to one another whereby agglomerates may be formed which would further assist in preventing movement or flowback of particulates from a wellbore or formation without significantly reducing the permeability of the particulate pack during aggressive flowback of treatment fluids.

[0012] We have now devised a method of treating a subterranean formation to form a porous particulate pack that inhibits the flow of particulates back through the wellbore during the production of hydrocarbons without significant effects upon the permeability of the particulate pack.

[0013] In accordance with the invention, a method of treating a subterranean formation penetrated by a wellbore is provided comprising the steps of providing a fluid suspension including a mixture of particulate material, a material comprising a liquid or solution of a tackifying compound, which coats at least a portion of the particulate upon admixture therewith, and a hardenable resin, which coats or is coated upon at least a portion of the particulate, pumping the fluid suspension including the coated particulate through the wellbore and depositing the mixture in the formation. Upon deposition of proppants having been coated with the tackifying compound and resin material mixture in the formation the coating causes particulate adjacent to the coated material to adhere to the coated material thereby creating proppant agglomerates which bridge against other particles in the formation to minimize initial particulate flowback and the hardenable resin subsequently consolidates the particulate before and during flowback.

[0014] The coated material is effective in inhibiting the flowback of particulate in a porous pack having a size ranging from about 2 to about 400 mesh in intimate admixture with the tackifying compound and hardenable resin coated particulates.

[0015] The coated material is effective in consolidating particulate into the form of agglomerates in a formation as a result of a fracturing or gravel packing treatment performed on a subterranean formation during aggressive flowback of the treatment fluid.

[0016] In accordance with the present invention, a liquid or solution of a tackifying compound is incorporated in an intimate mixture with a particulate material such as conventional proppants or gravel packing materials together with a hardenable resin and introduced into a subterranean formation.

[0017] As used in this specification, the term "intimate mixture" will be understood to mean a substantially uniform dispersion of the components in the mixture. The term "simultaneous mixture" will be understood to mean a mixture of components that are blended together in the initial steps of the subterranean formation treatment process or the preparation for the performance of the treatment process.

[0018] The coated particulate or proppant material may comprise substantially any substrate material that does not undesirably chemically interact with other components used in treating the subterranean formation. The material may comprise sand, ceramics, glass, sintered bauxite, resin coated sand, resin beads, metal beads and the like. The coated material also may comprise an additional material that is admixed with a particulate and introduced into a subterranean formation to reduce particulate flowback. In this instance the additional substrate material may comprise glass, ceramic, carbon composites, natural or synthetic polymers or metal and the like in the form of fibers, flakes, ribbons, beads, shavings, platelets and the like. In this instance, the additional substrate material generally will be admixed with the particulate in an amount of from about 0.1 to about 5 percent by weight of the particulate. The tackifying compound comprises a liquid or a solution of a compound capable of forming at least a partial coating upon the substrate material with which it is admixed prior to or subsequent to placement in the subterranean formation. In some instances, the tackifying compound may be a solid at ambient surface conditions and upon initial admixing with the particulate and after heating upon entry into the wellbore for introduction into the subterranean formation become a melted liquid which at least partially coats a portion of the particulate. Compounds suitable for use as a tackifying compound comprise substantially any compound which when in liquid form or in a solvent solution will form a non-hardening coating, by themselves, upon the particulate which facilitates agglomeration and will increase the continuous critical resuspension velocity of the particulate when contacted by a stream of water as hereinafter described in Example I by at least about 50 percent over the particulate alone when present in a 0.5 percent by weight active material concentration and increase the initial critical resuspension velocity by at least about 50 percent over the particulate alone. Preferably, the continuous critical resuspension velocity is increased by at least 100 percent over particulate alone and most preferably at least about 150 percent over particulate alone and the initial critical resuspension velocity is increased by at least 75 percent and most preferably at least 100 percent over particulate alone. A particularly preferred group of tackifying compounds comprise polyamides which are liquids or in solvent solution at the temperature of the subterranean formation to be treated such that the polyamides are, by themselves, non-hardening when present on the particulates introduced into

the subterranean formation. A particularly preferred product is a condensation reaction product comprised of commercially available polyacids and a polyamine. Such commercial products include compounds such as mixtures of C<sub>36</sub> dibasic acids containing some trimer and higher oligomers and also small amounts of monomer acids which are reacted with polyamines. Other polyacids include trimer acids, synthetic acids produced from fatty acids, maleic anhydride and acrylic acid and the like. Such acid compounds are available from companies such as Witco, Union Camp, Chemtall, and Emery Industries. The reaction products are available from, for example, Champion Chemicals, Inc. and Witco.

**[0019]** In general, the polyamides of the present invention are commercially produced in batchwise processing of polyacids predominately having two or more acid functionalities per molecule with a polyamine. As is well known in the manufacturing industry, the polyacids and polyfunctional amines are introduced into a reactor where, with agitation, the mildly exothermic formation of the amine salt occurs. After mixing, heat is applied to promote endothermic dehydration and formation of the polymer melt by polycondensation. The water of reaction is condensed and removed leaving the polyamide. The molecular weight and final properties of the polymer are controlled by choice and ratio of feedstock, heating rate, and judicious use of monofunctional acids and amines to terminate chain propagation. Generally an excess of polyamine is present to prevent runaway chain propagation. Unreacted amines can be removed by distillation, if desired. Often a solvent, such as an alcohol, is admixed with the final condensation reaction product to produce a liquid solution that can readily be handled. The condensation reaction generally is accomplished at a temperature of from about 225°F to about 450°F under a nitrogen sweep to remove the condensed water from the reaction. The polyamines can comprise, for example, ethylenediamine diethylenetriamine, triethylene tetraamine, amino ethyl piperazine and the like.

**[0020]** The polyamides can be converted to quaternary compounds by reaction with methylene chloride, dimethyl sulfate, benzylchloride, diethyl sulfate and the like. Typically the quaternization reaction would be effected at a temperature of from about 100 to about 200°F over a period of from about 4 to 6 hours.

**[0021]** The quaternization reaction may be employed to improve the chemical compatibility of the tackifying compound with the other chemicals utilized in the treatment fluids. Quaternization of the tackifying compound can reduce effects upon breakers in the fluids and reduce or minimize the buffer effects of the compounds when present in various fluids.

**[0022]** Additional compounds which may be utilized as tackifying compounds include liquids and solutions of, for example, polyesters, polyethers and polycarbamates, polycarbonates, natural resins such as shellac and the like.

**[0023]** The tackifying compound is admixed with the particulate in an amount of from about 0.1 to about 3.0 percent active material by weight of the coated particulate. It is to be understood that larger quantities may be used, however, the larger quantities generally do not significantly increase performance and could undesirably reduce the permeability of the particulate pack. Preferably, the tackifying compound is admixed with the particulate introduced into the subterranean formation in an amount of from about 0.25 to about 2.0 percent by weight of the coated particulate.

**[0024]** When the tackifying compound is utilized with another material that is to be admixed with the particulate and which is to be at least partially coated with the tackifying compound, such as glass fibers or the like, the compound is present in an amount of from about 10 to about 250 percent active material by weight of the glass fibers or other added material and generally from about 0.1 to about 3 percent active material by weight of the quantity of particulate with which the coated material is intimately admixed. Preferably the tackifying compound is present in an amount of from about 50 to about 150 percent of the material which is to be at least partially coated with the tackifying compound and then added to the particulate. At least a portion of the tackifying compound introduced with the additional material will contact and coat at least a portion of the particulate with which it is admixed.

**[0025]** The hardenable resin comprises an epoxy or phenolic resin or other compound capable of being at least partially coated upon a particulate substrate and then cured to a higher degree of polymerization. Examples of such resins include phenol-aldehyde resins of both the resole and novolac type, urea-aldehyde resins, melamine-aldehyde resins, epoxy resins, furfuryl alcohol resins and the like. The curing may result from heating the resin to a higher temperature such as can occur with the resole resins or by the addition of a catalyst or crosslinker to the resin which initiates polymerization. Admixtures of resins such as the resole and novolac resins may be utilized wherein sufficient resole resin is incorporated to initiate polymerization in the novolac resin. Various resins are described in for example U.S. Patents 5,420,174; 5,216,038; 5,425,994 and 4,888,240 the entire disclosures of which are incorporated herein by reference thereto. Particularly preferred resins include epoxy resins such as "EPON 828" epoxy resin from Shell Chemical Company, Houston, Texas. Phenolic resins such as "Resin 1866" from Acme Resin Corporation, Borden Division, Forrest Park, Illinois, furan resins such as "ARS-1500" resin from Advanced Resin Systems, Des Plaines, Illinois and novolac Resins such as "Bakelite 9282 FP" resin also available from Advanced Resin Systems.

**[0026]** The resin is admixed with the particulate in an amount of from about 0.01 to about 5.0 percent by weight of the particulate. Preferably, the resin is admixed with the particulate in an amount of from about 0.05 about 1.0 percent by weight of the particulate, Curing agents, catalysts or crosslinkers selected from those well known in the art may be utilized with the resin to harden the resin and form a consolidated matrix of particulate.

**[0027]** The liquid or solution of tackifying compound interacts mechanically with the particles of particulate introduced

into the subterranean formation to limit or prevent the flowback of particulates to the wellbore during initial flowback prior to hardening and consolidation of the particulates by the hardenable resin.

[0028] In one embodiment, the tackifying compound, when comprised of polyamides that contain reactive sites such as amine groups, may be admixed and contacted with a material that has multi-functional reactive sites which are capable of reacting with the reactive sites on the tackifying compound to form a hard reaction product which consolidates the agglomerates formed by the tackifying compound. A "hard reaction product" as used herein means that the reaction of the tackifying compound with the multifunctional material will result in a substantially nonflowable reaction product that exhibits a higher compressive strength in a consolidated agglomerate than the tackifying compound alone with the particulates. One means of evaluating the consolidated agglomerate to determine whether an increase in compressive strength has occurred is through testing with a penetrometer. Samples may be prepared comprising particulate coated with the tackifying compound, multifunctional material and an admixture as described herein and penetrometer readings can be made using equipment such as a PWG Penetrometer from Precision Scientific Company, Chicago, Illinois. Comparison of the penetrometer readings readily demonstrates the change that has occurred as a result of the reaction. In this instance, the tackifying compound also functions as the hardenable resin. The material having multi-functional reactive sites include compounds such as aldehydes such as formaldehyde, dialdehydes such as glutaraldehyde, hemiacetals or aldehyde releasing compounds, diacid halides, dihalides such as dichlorides and dibromides, polyacid anhydrides such as citric acid anhydride, epoxides and the like. Preferred compounds for use with polyamides containing reactive sites comprise furfuraldehyde, glutaraldehyde or aldehyde condensates and the like. The multi-functional compound is admixed with the tackifying compound in an amount of from about 0.01 to about 50 percent by weight of the tackifying compound to effect formation of the reaction product. Preferably, the compound is present in an amount of from about 0.5 to about 1 percent by weight of the tackifying compound.

[0029] The liquid or solution of tackifying compound and hardenable resin generally are incorporated with the particulate in any of the conventional fracturing or gravel packing fluids comprised of an aqueous fluid, an aqueous foam, a hydrocarbon fluid or an emulsion, a viscosifying agent and any of the various known breakers, buffers, surfactants, clay stabilizers or the like.

[0030] Generally the tackifying compound and hardenable resin may be incorporated into fluids having a pH in the range of from about 3 to about 12 for introduction into a subterranean formation. The compounds are useful in reducing particulate movement within the formation at temperatures from about ambient to in excess of 300°F. It is to be understood that not every hardenable resin or tackifying compound will be useful over the entire pH or temperature range but every compound is useful over at least some portion of the range and individuals can readily determine the useful operating range for various products utilizing well known tests and without undue experimentation.

[0031] The liquid or solution of tackifying compound and the hardenable resin generally are incorporated with the particulate as a simultaneous mixture by introduction into the fracturing or gravel packing fluid along with the particulate. Fracturing fluid slurries are introduced into the subterranean formation at a rate and pressure sufficient to create at least one fracture in the formation into which particulate then is introduced to prop the created fracture open to facilitate hydrocarbon production. Gravel packing treatments generally are performed at lower rates and pressures whereby the fluid can be introduced into a formation to create a controlled particle size pack surrounding a screen positioned in the wellbore where fracturing of the formation may or may not occur. The particulate pack surrounding the wellbore then functions to prevent fines or formation particulate migration into the wellbore with the production of hydrocarbons from the subterranean formation.

[0032] The gravel packing treatment also may be performed without a screen in the wellbore. In such a screenless completion, the fluid generally is introduced into the wellbore to fill the perforations and wellbore to a level above the perforations and permitted to consolidate. The consolidated pack can then be drilled or reamed out to reopen the bore while providing a consolidated pack to screen fines and formation particulate from migrating into the wellbore.

[0033] The tackifying compound may be introduced into the fluid before, after or simultaneously with introduction of the particulate into the fluid. The liquid or solution may be incorporated with the entire quantity of particulate introduced into the subterranean formation or it may be introduced with only a portion of the particulate, such as in the final stages of the treatment to place the intimate mixture in the formation in the vicinity of the wellbore. For example, the tackifying compound may be added to only the final 20 to 30 percent of the particulate laden fluid introduced into the formation and the hardenable resin may be added to only the last 10 to 20 percent of the particulate laden fluid. In this instance, the intimate mixture will form a tail-in to the treatment which upon interaction within the formation with the particulate will cause the particles to bridge on the agglomerates formed therein and prevent movement of the particles into the wellbore with any produced fluids. The tackifying compound and hardenable resin may be introduced into the blender or into any flowline in which they will contact the material to be at least partially coated by the compounds. The compounds may be introduced with metering pumps or the like prior to entry of the treatment fluid into the subterranean formation.

[0034] In an alternate embodiment, the particulate may be premixed with either the tackifying compound or the hardenable resin prior to admixing with a treatment fluid and the other constituents for use in a subterranean formation. In

some instances, resin precoated particulates may be utilized and the tackifying compound then would be added during performance of the subterranean formation treatment. Depending upon the type of resin coating employed, a catalyst then would be added to the treatment fluid or introduced in a flush fluid or the like.

[0035] Surprisingly, it has been found that use of the method of the present invention can produce high permeability tunnels extending from wellbore perforations back into proppant packed fractures created in the subterranean formation which then may be consolidated. Control of the flowback rate of the treatment or formation fluids from the wellbore can be used to provide a controlled erosion of the treated particulate immediately adjacent a perforation in the wellbore. The flowback rate is controlled so as to provide a level above the initial critical resuspension velocity of the tackifying compound but generally is maintained below the continuous critical resuspension velocity. This results in controlled production of particulate from the formation. The erosion surprisingly has been found to be very uniform in nature and to create a tunnel into the particulate in the formation generally corresponding to the size and shape of the perforation in the wellbore. After the tunnel is formed, the hardenable resin consolidates the remaining particulate to provide a high permeability passage or tunnel from the formation to the wellbore.

[0036] To further illustrate the present invention and not by way of limitation, the following examples are provided.

#### EXAMPLE I

[0037] The evaluation of a liquid or solution of a compound for use as a tackifying compound is accomplished by the following test. A critical resuspension velocity is first determined for the material upon which the tackifying compound is to be coated. The apparatus comprises a 1/2" glass tee which is connected to an inlet source of water and an outlet disposal line is blocked to fluid flow. A water slurry of particulate is aspirated into the tee through the inlet and collected within a lower portion of said tee by filtration against a screen. When the lower portion of the tee is full, the vacuum source is removed and a plug is used to seal the end of the lower portion of the tee. The flow channel from inlet to outlet then is swabbed clean and a volumetrically controlled pump, such as a "MOYNO" pump, is connected to the inlet and a controlled flow of water is initiated. The velocity of the fluid is slowly increased through the inlet until the first particle of particulate material is picked up by the flowing water stream. This determines the baseline for the starting of the resuspension velocity. The flow rate then is further increased until the removal of particles becomes continuous. This determines the baseline for the continuous resuspension velocity. The test then is terminated and the apparatus is refilled with particulate having a coating corresponding to about 0.5 percent active material by weight of the particulate applied thereto. Similar trends generally are seen in the results when the concentrations tested are from about 0.1 to about 3 percent, however, the 0.5 percent level which is within the preferred application range is preferred for standardization of the procedure. The test is repeated to determine the starting point of particulate removal and the velocity at which removal becomes continuous. The percent of velocity increase (or decrease) then is determined based upon the initial or continuous baseline value. The results of several tests employing the preferred polyamide of the present invention, and conventional epoxy and phenolic resins known for use in consolidation treatments in subterranean formations with 12/20 and 20/40 mesh sand are set forth below in Table I.

TABLE I

Test No.	Particulate Size	Coating Agent, % V/Wt Particulate	Percent Of Velocity Change At:	
			Starting of Sand Particle Transport	Continuous Sand Transport
1	20/40/mesh sand	None	0	
2	20/40 mesh sand	1/2 percent polyamide	192	222
3	20/40 mesh sand	1 percent polyamide	271	391
4	20/40 mesh sand	1/2 percent phenolic	-0.5	6.5
5	20/40 mesh sand	1 percent phenolic	-9	-6.3
6	20/40 mesh sand	1/2 percent epoxy	-9	-1.2
7	20/40 mesh sand	percent epoxy	5.2	12.2
6	12/20 mesh sand	1/2 percent polyamide	228	173
9	12/20 mesh sand	1 percent polyamide	367	243
10	12/20 mesh sand	1/2 percent phenolic	42	22
11	12/20 mesh sand	1 percent phenolic	42	13

TABLE I (continued)

Test No.	Particulate Size	Coating Agent, % V/Wt Particulate	Percent Of Velocity Change At:	
			Starting of Sand Particle Transport	Continuous Sand Transport
12	12/20 mesh sand	1/2 percent epoxy	43	30
13	12/20 mesh sand	1 percent epoxy	38	15

**[0038]** The data clearly illustrates the substantial increase in the critical resuspension velocity of a particulate coated with the tackifying compound in comparison to other known formation consolidation agents which require hardening to be effective.

**[0039]** The test results clearly demonstrate the beneficial results achieved by practice of the method of the present invention with respect to proppant production from a simulated formation.

#### EXAMPLE II

**[0040]** The stabilization properties of the method of the present invention are determined by comparison to untreated sand and sand including a tackifying compound. The flowback velocity is measured in an American Petroleum Institute approved simulated fracture flow cell. The cell contains Ohio sandstone cores having a proppant bed size of about 1.5 inches in height, about 7 inches in length and about 0.25 inches in width between the cores. The bed is initially pre-packed with 20/40 mesh sand by introducing the sand into the cell in an aqueous slurry or a gelled fluid containing 40 pounds of guar per 1000 gallons of aqueous fluid. The cell is fitted with a 0.3 inch hole at one end to simulate a perforation. The hole is visible through a sight glass so that proppant production through the hole can be visually determined.

**[0041]** The cell then was cleaned and packed with another proppant pack for testing. The tested materials are set forth in Table II, below.

TABLE II

SAMPLE	PACKING FLUID	ADDITIVES TO SAND, % BY WT. SAND	FLOW RATE, ml/min AT WHICH FAILURE OCCURS
1	water	None	84
2	gel	None	90
3	gel	1% by wt polyamide	180
4	gel	2% by wt polyamide	384
5	gel	1% by wt polyamide and 1% out Bakelite 9282 FP resin	>3000 <sup>1</sup>
6	gel	1% by wt polyamide and 1% by wt Bakelite 9282 FP resin	>2600 <sup>2</sup>

<sup>1</sup>. pack heated at 250°F for 72 hours before testing, no sand production during test

<sup>2</sup>. pack heated at 180°F for 4 hours before testing no sand production during test

#### Claims

1. A method of treating a subterranean formation in a well, which method comprises the steps of contacting the formation with a treatment fluid comprising a particulate, a liquid or solution of a tackifying compound comprising a polyamide, at least a portion of said particulate being at least partially coated by said compound such that the critical resuspension velocity of said at least partially coated particulate is increased by at least 50 percent when tested at a level of 0.5% active material by weight over said particulate alone with water; and a multifunctional material to react with at least a portion of said tackifying compound on said particulate to form a hard reaction product; depositing the tackifying compound-coated particulates in the subterranean formation; and flowing back fluid from the formation, the tackifying compound coated particulate retarding movement of at least a portion of the particulate within said formation and said multifunctional material causing a hard reaction product to form that subsequently consolidates at least a portion of the particulate within said formation.

2. A method according to claim 1, wherein the treatment fluid is formed by mixing some or all of the components downhole.
- 5 3. A method according to claim 1, wherein the treatment fluid is formed before introducing it into the well.
4. A method according to claim 1, 2 or 3, wherein said tackifying compound is mixed with said particulate in an amount of from 0.1 to 3.0 percent by weight of said particulate.
- 10 5. A method according to claim 4, wherein said tackifying compound is mixed with said particulate in an amount of from 0.25 to 2 percent by weight of said particulate.
6. A method according to any of claims 1 to 5, wherein said coated particulate has a critical resuspension velocity in excess of 100 percent over said particulate alone.
- 15 7. A method according to any of claims 1 to 6, wherein said polyamide comprises predominantly a condensation reaction product of a dimer acid containing some trimer and higher oligomers and some monomer acids with a polyamine.
- 20 8. A method according to claim 7, wherein said polyamine comprises at least one of ethylenediamine, diethylenetriamine, triethylenetetraamine, tetraethylene pentaamine and aminoethylpiperazine.
- 25 9. A method according to any of claims 1 to 8, wherein said multifunctional material comprises at least aldehyde, dialdehyde, diacid halide, dihalide, polyacid anhydride, epoxide or hemiacetal, or any mixture of two or more thereof.
- 30
- 35
- 40
- 45
- 50
- 55



# BEST AVAILABLE COPY

EP 1 001 133 A1



European Patent  
Office

## EUROPEAN SEARCH REPORT

Application Number  
EP 99 30 7988

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.Cl.7)
X	EP 0 853 186 A (HALLIBURTON ENERGY SERV INC) 15 July 1998 (1998-07-15) * page 3, line 14 - line 31 * * page 5, line 2 - line 19 * * page 5, line 43 - line 56 *	1-9	E21B43/26 E21B43/267 E21B43/04
Y	US 5 775 425 A (PARKER MARK A ET AL) 7 July 1998 (1998-07-07) * column 3, line 24 - line 57 * * column 4, line 32 - column 5, line 3 * * column 5, line 66 - column 6, line 9 *	1-5,7-9	
Y	EP 0 834 644 A (HALLIBURTON ENERGY SERV INC) 8 April 1998 (1998-04-08) * page 3, line 25 - page 4, line 16 * * page 4, line 40 - line 43 * * page 5, line 5 - line 24 *	1-9	
Y	EP 0 859 125 A (HALLIBURTON ENERGY SERV INC) 19 August 1998 (1998-08-19) * page 3, line 49 - page 4, line 12 * * page 4, line 44 - line 48 *	1-9	
			TECHNICAL FIELDS SEARCHED (Int.Cl.7)
			E21B
The present search report has been drawn up for all claims			
Place of search <b>THE HAGUE</b>		Date of completion of the search <b>15 February 2000</b>	Examiner <b>Boulon, A</b>
<p><b>CATEGORY OF CITED DOCUMENTS</b></p> <p>X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document</p> <p>T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons</p> <p>&amp; : member of the same patent family, corresponding document</p>			

EPO FORM 1503 03.82 (p.04/01)

**ANNEX TO THE EUROPEAN SEARCH REPORT  
ON EUROPEAN PATENT APPLICATION NO.**

EP 99 30 7988

This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report. The members are as contained in the European Patent Office EDP file on. The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

15-02-2000

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
EP 0853186 A	15-07-1998	US 5839510 A	24-11-1998
		BR 9708116 A	05-10-1999
		CA 2217637 A	14-07-1998
		NO 974554 A	15-07-1998
US 5775425 A	07-07-1998	US 5787986 A	04-08-1998
		US 5582249 A	10-12-1996
		US 5501274 A	26-03-1996
		BR 9706876 A	29-06-1999
		CA 2217638 A	19-11-1998
		EP 0879935 A	25-11-1998
		NO 974556 A	20-11-1998
		US 5960878 A	05-10-1999
		US 5853048 A	29-12-1998
		US 5871049 A	16-02-1999
		BR 9706874 A	15-06-1999
		CA 2217627 A	03-04-1998
		EP 0834644 A	08-04-1998
		NO 974553 A	06-04-1998
		US 5839510 A	24-11-1998
		US 5833000 A	10-11-1998
EP 0834644 A	08-04-1998	EP 0735235 A	02-10-1996
		NO 953109 A	30-09-1996
EP 0834644 A	08-04-1998	US 5787986 A	04-08-1998
		BR 9706874 A	15-06-1999
		CA 2217627 A	03-04-1998
		NO 974553 A	06-04-1998
		US 5960878 A	05-10-1999
		US 5853048 A	29-12-1998
		US 5871049 A	16-02-1999
		US 5839510 A	24-11-1998
		US 5833000 A	10-11-1998
		US 5775425 A	07-07-1998
EP 0859125 A	19-08-1998	US 5833000 A	10-11-1998
		BR 9706875 A	28-09-1999
		CA 2217636 A	18-08-1998
		NO 974555 A	19-08-1998

EPO FORM P0459

For more details about this annex : see Official Journal of the European Patent Office, No. 12/82